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Levels and environmental impact of PAHs and trace element in fly ash from a miscellaneous solid waste by rotary kiln incinerator, China

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Abstract The increase disposals of fly ash from solid waste incinerator have becoming a serious environmental problem in China. Levels of PAHs in fly ash from a selected full-scale miscellaneous solid waste incinerator were determined by gas chromatograph/mass spectrometer. Content of trace elements and other major elements of fly ash were studied by inductively coupled plasma-mass spectrometry and X-ray fluorescence spectrometry, respectively. Experimental results showed that a high content of lead occurred in the collected fly ash samples with the value of 35,037.90 µg/kg, but lower contents of total 16 PAHs, which mainly smaller PAHs. This was different from previous studies regarding the levels of PAHs in fly ash collected from the similar hospital waste incinerators. This survey indicated that the emission factors are attributed to combustion temperatures, type of incinerator, and the occurrences of elements in solid wastes. The total toxic equivalent quantities (TEQ) value of PAHs, a potential toxicity risk assessment using index TEQ model, has been reached to 4.00 µg/kg in ash from this study, but may not suitable for the soil amendment. Therefore, a further treatment of discharged ash and assessment of its environmental risk are unavoidable.

Keywords Environmental impact · PAHs · Fly ash · Hazardous solid waste

1 Introduction

China, one of the fastest developing countries, is the world's largest solid waste generator due to rapid industrialization and urbanization in the last two decades (Duan et al. 2008;

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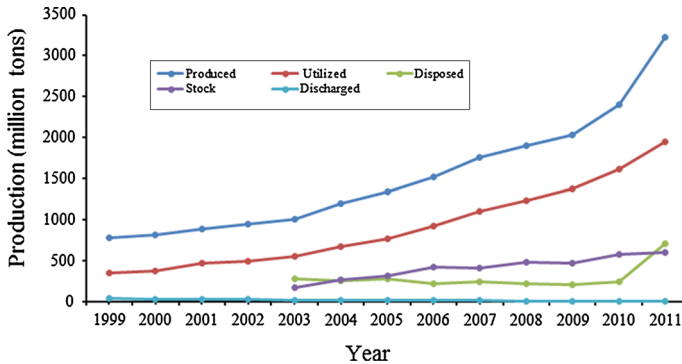
Fig. 1 Situation of industrial solid wastes in China from 1999 to 2011

Huang et al. 2006; Zhang et al. 2010). Take the industry solid waste (ISW), for example, the total quantity has increased alarmingly year by year, which increased from 0.8 billion tons in 1998 to 3.2 billion tons in 2011, according to the statistical records of the ISW (China Statistical Yearbook 1998–2011). Statistic quantity of ISW was about 3.3 billion tons in 2012 (Chinese Environmental Yearbook 2012). At present, more than half of total ISW is reused in China (Fig. 1), while there is still vast majority of ISW-treated stock subsequently discharged into the environment. Solid waste crisis is serious in China (Liu et al. 2006; Tian et al. 2013). Due to the limited land available for landfills, incineration is safe and effective option for waste management as it could significantly reduce the volume and weight of waste and destroy pathogens and toxic organic compounds (Liu et al. 2000b). However, fly ash is inevitable during waste incineration, accounting for 10–30 wt% of the original amount of waste (Tian et al. 2012; Zhang et al. 2008). Most of them are characterized by high enrichment of toxic chemicals such as trace elements and organic pollutants (Koukouzas et al. 2011; Tang et al. 2013; Wang et al. 2010). These pollutants are likely to return into the environment and cause secondary pollution and thus into the food web, if improper management (Dolk et al. 1998). For this reason, fly ash is seen as hazardous materials and needs to specially manage in many countries (Haugsten and Gustavson 2000). Polycyclic aromatic hydrocarbons (PAHs), one typical persistent toxic substance, consist of 2–6 condensed aromatic rings (Directive 2000), which are one of the main organic contaminants in fly ash particle that have been added into the list of the priority pollutants and the emission standards by the World Health Organization, as well as America, France, Germany, Japan, Netherlands, Sweden, and Switzerland (Wilson and Jones 1993; OECD 1989; Environment Australia 1999; World Health Organization 1985; WHO 2000; Wilson and Jones 1993). Large amount of study about the PAH characteristics focused on coal, municipal solid wastes (MSW), biomass, industrial hazardous, and medical wastes (Bozlaker et al. 2008; Johansson and van Bavel 2003b; Lee et al. 2002; Liu et al. 2001; McGrath et al. 2001). To the best of authors' knowledge, however, only limited data are available in the research about the PAHs in the fly ash from the miscellaneous waste combustion. In addition, no official standards have been issued for PAHs in the fly ash and quite few contamination prevention measures have been made for fly ash in China. Therefore, it is necessary to investigate the PAH concentrations in miscellaneous waste combustion to fill this gap. In the present work, we address this aspect by discussing the characteristics of fly ash from a miscellaneous solid waste incinerator in China.

2 Samples and methods

2.1 Basic information on incinerator and sampling

The investigation was carried out in a rotary kiln incinerator located in the east of China. The incinerator began operation during the outbreak of severe acute respiratory syndrome (SARS) in 2003 with the capacity of 6,000 t/year solid waste (Table 1). Solid waste incinerated is very heterogeneous and mainly consists of hospital waste (HW; 65 %) and industries hazardous waste (35 %). The incinerator is equipped with an air pollution control device of bag filter as shown schematically in Fig. 2. To obtain representative samples, fly ash samples were taken directly from the hopper of the bag filter when the system tuning to a stable condition on five successive days; then, five fly ash samples were kept in vacuum-sealed bags before analysis.

2.2 Methods

All the samples for trace element analysis were crushed and ground to pass a 200-mesh sieve after dried at 105 °C for 24 h. Physicochemical parameters including particle size distribution and specific surface were determined by laser diffraction particle size analyzer (Better BT 2003) and nitrogen adsorption–desorption isotherms, respectively.

Content of trace elements in fly ash was determined by inductively coupled plasma-mass spectrometry (ICP-MS) after digestion by an acid mixture of $HNO_3:HCl:HF$ (3:1:1) in closed vessels with microwave oven. The chemical composition was determined by

Table 1 Operational conditions and parameters of the incinerator

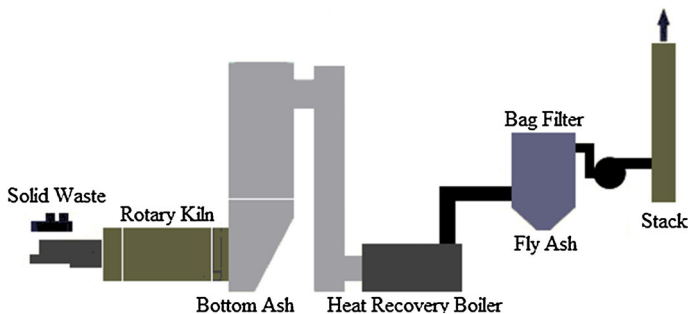| Operation beginning year	2003
Operation (h/day)	24
Type	Rotary kiln incinerator
Annual capacity (t/year)	6,000
Furnace temperature (°C)	First combustion chamber: 600–800
	Second combustion chamber: 850–1,000
Input waste	Combustible industrial waste 35 %
	Medical waste 65 %
APC devices	Fabric filters

Fig. 2 Schematic diagram of the solid waste incinerator

X-ray fluorescence spectrometry (XRF) following the method ASTM D4326-2001. The accuracy of the analytical method was determined by analyzing standard reference material NBS1633b. The recovery rates of the trace element were within the certificate values.

Method for PAH analysis is consistent with the previous analysis process, which has been described previously (Liu et al. 2012). Samples were dried at 30 °C for 24 h, crushed and sieved through a 200-mesh sieve. About 15 g of pretreated sample was extracted in a Soxhlet extractor with 250 mL dichloromethane for 48 h. Two grams of activated copper piece was added to remove sulfur. The extract was preconcentrated to a volume of 1 mL by rotary evaporation, and its solvent was exchanged for hexane. The concentrated solution (~ 2 mL) was injected to pass through a purifying tube packed with silica gel and neutral alumina. After eluting with 70 mL of the methylene dichloride-hexane (3:7 v/v) mixture, the PAH fractions were concentrated to 1.5 mL under a gentle stream of N_2.

Determination of PAHs was conducted on a Shimadzu model 2010 GC–MS instrument equipped with an AOC-20i autoinjector (Shimadzu, Japan), using electron impact mode (70 eV) in the selected ion monitoring mode. The DB-5 column of 60 m × 0.25 mm inner diameter (0.25 μm film thickness) was used. The GC temperature increased from 60 to 200 °C at 5 °C/min, to 250 °C at 2 °C/min, and then to 290 °C at 20 °C/min and further hold for 20 min at 290 °C. The carrier gas Helium (99.9999 % purity) was used as carrier gas at a constant flow rate of 1.0 ml/min.

The quantitative analysis was done by the internal calibration method (five-point calibration), and PAH identification was performed by comparison of their retention time with standards. A mixture of internal standards 2-fluoro-1, 1-biphenyl, p-terhenyl-d14 and dibenzo(a,h)anthracene-d14 was added to the sample just before injection. 2-Fluoro-1 and 1-biphenyl were used for Naph, Acy, Acen, and Flu; p-terhenyl-d14 was used for Phen, Anth, Flan, Pyr, B[a]A, Chry, B[b]F, B[k]F, and B[a]P; dibenzo(a,h)anthracene-d 14 was used for IP, DB[ah]A, and B[ghi]P.

Reagent blanks, duplicate samples, and the standard additions were analyzed along with samples through the procedures of extraction and cleanup. Reagent blank samples contained no detectable amounts of the monitoring PAHs. Surrogate standards containing five deuterated PAHs (naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12) were added to all samples before extraction to determine the recovery rate of the analysis process. The surrogate recoveries were 67.2 ± 4.52 % for naphthalene-d8; 75.4 ± 4.67 % for acenaphthene-d10; 88.1 ± 6.41 % for phenanthrene-d10; 94.8 ± 4.35 % for chrysene-d12; and 98.5 ± 6.59 % for perylene-d12 with fly ash samples. Meanwhile, recoveries of 16 PAHs in the NIST1941 reference sample were between 80 and 120 % of the certified values provided by the NIST.

3 Results and discussion

3.1 Physiochemical properties of collected fly ash

The particle size was determined by laser diffraction particle size analyzer (Better BT 2003), and the result was given in Fig. 3. The particle sizes of most ash were within a range of 0.079–30.67 μm, accounting for more than 90 wt% of the total ash mass. The average particle size was 9.85 μm. Similar ranges of ash particle size have been found in previous studies. For example, Cobo et al. (2009) reported that about 75 wt% of the total mass of fly ash collected from the bag house of a hazardous waste incinerator. Nagib and Inoue (2000) found about 80 wt% of the total mass of fly ash from the MSW incinerator has particle size
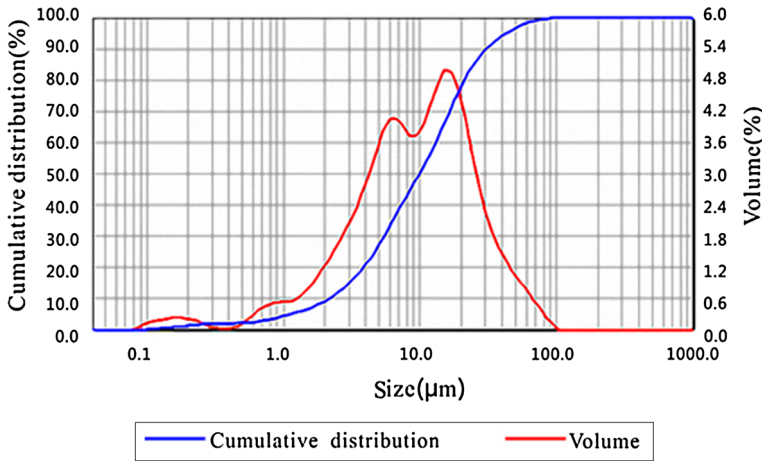
Fig. 3 Typical particle size distribution of the fly ash

<20 μm. Moreover, the fly ash is characterized by tri-modal particle size distribution, which includes a smallest ultrafine region centered at approximately 0.2 μm, a fine fragmentation centered at 8 μm, and a bulk fragmentation region for particles of approximately 15 μm diameter. The smallest fly ash particle was formed mainly by solid–vapor–particle processes, including vaporization, nucleation, condensation, and coagulation of inorganic constituents in the waste (Xu et al. 2011). The central mode may attribute to the coagulated fine particles or micrometer-size fuel materials (Yu et al. 2009). Fusion and coalescence of the inorganic material in the fuel should be mainly responsible for the coarse-mode formation.

The nitrogen adsorption–desorption isotherms of this fly ash are shown in Fig. 4. Based on the IUPAC classification, the adsorption isotherms of this ash attribute to the Type II adsorption, indicating a presence of micropores (larger than 2 nm). As presented in Table 2, the S_{BET} of ash particle size was found to be 3.92 m^2/g and also suggests the porosity of particles.

The chemical composition of ash samples was analyzed using XRF, and the result was illustrated in Table 3; SiO_2 was the most abundant component in ash sample followed by Al_2O_3 and Fe_2O_3. The least abundant major components were MnO, SO_3, and P_2O_5. Higher content of SiO_2 makes a more fixed structure to restrain trace element from leaching (Song et al. 2004).

3.2 Trace element in fly ash

Although trace elements have much less proportions in view of the unit mass of fly ash, the large amount production of fly ash from solid waste incinerator will significantly increase amounts and risks of the released trace elements into the environment. This generally occurs during the landfilling disposal under natural conditions. The mean content of selected 10 trace elements in fly ash is shown in Table 3. Higher contents of volatile trace elements were found to be bound to the collected ash that was consistent with the results reported in previous works (Cobo et al. 2009; Eighmy et al. 1995). Especially, lead is an element with the highest content in all the samples in this study, and its content in the ash
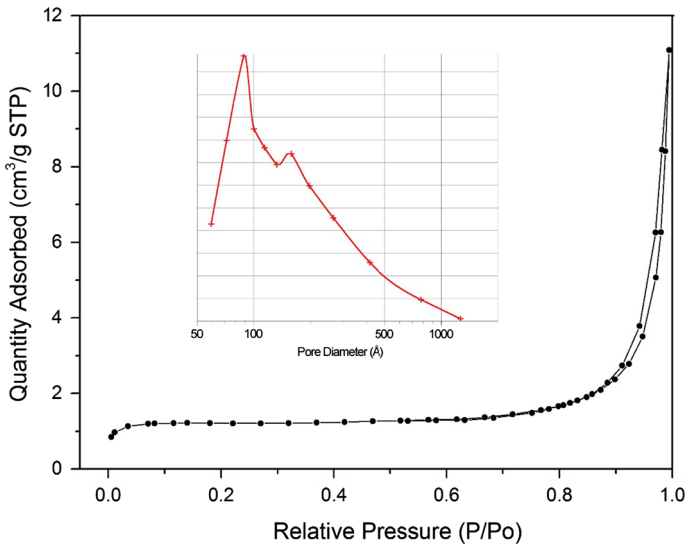
Fig. 4 Adsorption–desorption isotherms of fly ash

Table 2 Particle size distribution of size fractionated fly ash

Undersize (%)			D_{10} (μm)	D_{50} (μm)	D_{90} (μm)	AMD (μm)	Surface area (m²/g)
<10 μm	<50 μm	<100 μm					
50.58	96.91	100.00	2.15	10.02	31.03	15.65	0.53

D_{10} 10 % of the particles is smaller than this diameter (μm); D_{50} 50 % of the particles is smaller than this diameter (μm); D_{90} 90 % of the particles is smaller than this diameter (μm); *AMD* average mean diameter (μm)

Table 3 Content of major and trace element in fly ash

Major elements	Fe_2O_3	MnO	TiO_2	CaO	K_2O	SO_3	P_2O_5	SiO_2	Al_2O_3	Na_2O	MgO	Total
Fly ash (%)	4.72	0.099	10.904	8.27	0.83	1.74	0.325	47.66	6.39	8.03	1.32	90.29

Trace elements	Co	Ni	Mn	Pb	Cr	Cd	As	B	Cu	V
Fly ash (μg/kg)	25.92	24.11	562.6	35,037.90	260.70	2.94	4.90	24.71	2,918.6	3.90

reaches 35,037.90 μg/kg. This may due to the different physicochemical properties of elements and their content in the solid waste (Chang et al. 2009). Increasing the combustion temperatures, some low-boiling point trace elements, such as Zn, Pb, Cd, and Cu, preferably volatilized in combustion zone and formed the uniform small particles and then condensed on the fly ash in the decreasing temperature flue gas convective pass to stack (Song et al. 2004). In China, there has not been setup of regulation in trace elements in fly ash from specific HWs. Based on the available standard issued by the California EPA,

which is 5,000 µg/kg, the content of Pb in the current fly ash far more exceeds this regulatory thresholds. Therefore, fly ash in the incinerator should be properly disposed to reduce its environmental risks; otherwise, it may affect humans by the food chain.

3.3 Content of PAHs in fly ash

PAHs are a group of semi-volatile organic compounds that consist of 2–6 condensed aromatic rings (Kong et al. 2011; Sun et al. 2006; Yang et al. 2002). Many of these compounds are known as carcinogenic, mutagenic, and teratogenic. Some PAHs adsorbed on ash can cause serious environmental pollution and can persistent in the environment for months due to their stable structure if they evaporate into the atmosphere (Liu et al. 2000a, 2008). That's why the US Environmental Protection Agency has issued a list of priority pollutants to be monitored in the environment, including 16 PAHs according to their toxicity (Callahan 1979; Ravindra et al. 2001).

The total content and individual contents of the 16 USEPA priority PAHs found in the fly ash samples are summarized in Table 4. The results showed that the total content of PAHs was 2,444.90 µg/kg, PAHs of five to six rings (Big PAHs) were not detected, and the content of most toxic PAH (B[a]P) was not found. PAHs with two to four rings (small

Table 4 Concentrations of PAHs in fly ash samples (µg/kg)

Congener (µg/kg)	Fly ash (N = 5)		TEF[a]	TEQ
	Mean ± SD	Range		
Naphthalene	1,940.64 ± 7.40	1,930.36–1,949.23	0.001	1.941
Acenaphthylene	143.94 ± 12.71	124.11–154.21	0.001	0.144
Acenaphthene	46.05 ± 8.31	33.60–54.77	0.001	0.046
Fluorene	42.39 ± 11.74	33.80–65.40	0.001	0.042
Phenanthrene	49.81 ± 3.71	44.31–53.70	0.001	0.050
Anthracene	42.07 ± 3.81	40.05–49.12	0.01	0.421
Fluoranthene	57.04 ± 6.86	44.16–59.70	0.001	0.057
Pyrene	57.65 ± 5.60	50.58–65.03	0.001	0.058
Benz[a]anthracene[b]	3.83 ± 0.66	3.17–4.92	0.1	0.383
Chrysene[b]	58.71 ± 5.76	50.15–63.58	0.01	0.587
Benzo[b]fluoranthene[b]	1.38 ± 0.50	0.93–2.19	0.1	0.139
Benzo[k]fluoranthene[b]	1.34 ± 0.51	1.22–2.32	0.1	0.134
Benzo[a]pyrene[b]	ND	ND	1	0.000
Indeno[123-cd]pyrene[b]	ND	ND	5.000	0.000
Dibenz[a,h]anthracene[b]	ND	ND	0.001	0.000
Benzo[g,h,i]perylene	ND	ND	0.100	0.000
ΣPAHs[c]	2,444.85			4.002
ΣPAHs$_{carc}^{d}$	65.26			

ND not detectable

[a] PAHs toxic equivalency factor with respect to BaP (Nisbet and LaGoy 1992)

[b] Carcinogenic PAH compounds

[c] Sum concentration of 16 PAH compounds

[d] Sum concentration of 7 carcinogenic PAH compounds

PAHs) have been identified in fly ash samples. Naphthalene is the most abundant PAH present in samples with a content of 1,940.64 µg/kg. The content of the 7 USEPA carcinogenic PAHs ($\Sigma PAHs_{carc}$: BaA, CHR, BbF, BkF, BaP, IcdP, and DahA) was 65.28 µg/kg and accounted for 2.7 % of the total PAH content in mass.

Emission of PAHs from solid waste was determined by the combustion conditions of incinerators, mainly referring to temperatures, retention times, and incinerator structures (Singh and Prakash 2007). In a low-temperature stage (below 800 °C), the major pathway for the formation of PAHs is pyrolysis of solid wastes, resulting in the formation of small PAHs. While the large PAHs are produced during the deep degradation and/or combination of small PAHs in a higher temperature (Wheatley and Sadhra 2004; Yan et al. 2004; Zhao et al. 2000), study of Singh and Prakash (2007) acclaimed the highest content of total PAHs in fly ash at 1,000 °C.

As for the current rotary kiln incinerator, lower combustion temperature was applied (850 °C) in the first combustion chamber. This may partly explain better prevention of PAHs during combustion of solid wastes. Studies of Van Caneghem et al. (2010, 2014) were shown that the PCBs (polychlorinated biphenyls) in rotary kiln have been destroyed effectively during incineration by mass balance method. Li et al. (2004) also confirmed the increase of PAHs content, especially small PAHs, upon the increase of combustion temperature. Thus, he suggested two-stage combustion of solid wastes. Temperature in the first combustion chamber was about 800 °C, and it was followed up by the second combustion chamber, controlling the temperature at about 1,000 °C and residence time at 2 s for a complete combustion of the vaporizing PAHs from the first chamber. The basic procedure occurrence of PAHs generated in ash, as shown in Table 4. Small PAHs have higher stability and can travel over a longer distance due to their higher boiling point comparing to large PAHs (Chen et al. 2003; Lai et al. 2007; Liu et al. 2000a; Tsai et al. 2002). Because of this, small PAHs usually cause higher risks than big PAHs although they have little carcinogenicity effect.

Comparing with the earlier study by Wild et al. (1992), it was found the amount of 16 PAHs in fly ash from coal combustion and MSW incinerators was 354 and 227 µg/kg, respectively. Results of similar studies (Chen et al. 2003; Lee et al. 2002; Levendis et al. 2001; Zhao et al. 2008) of PAH contents in fly ash from HW incinerators are shown in Fig. 5. The content of PHAs in fly ash in this study was higher than in coal fly ash and MSW fly ash and much lower than in HW fly ash in other studies. This difference can possible interpreted as the higher calorific value of solid waste in this study with more content of plastic, accounting for 13–18 % in medical waste (Chen et al. 2013; Fang et al. 2005).

Fig. 5 Comparison of 16 PAH contents in fly ashes from hospital waste (HW) incinerators between this study and the literature (µg/kg)
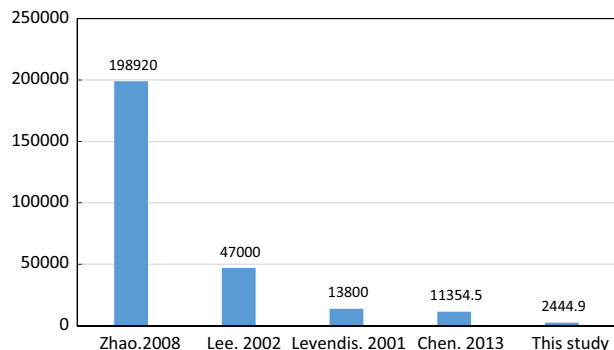
Another factor that may cause the high PAHs in the ash is the inherently higher PAH content in the waste components (Wheatley and Sadhra 2004). Meanwhile, the presence of Pb in fly ash may facilitate the formation of PAH, which have been discussed by Wey et al. (2000).

3.4 Potential toxicity risk assessment for PAHs in fly ash

Since no environmental standards for PAHs in ash are established in China, standards developed in Swedish and Dutch were adopted to evaluate the PAH contamination. Guidelines for PAHs in land-use limit have been developed by Swedish EPA, Netherland EPA, and Canada EPA, and for sensitive land use, the limits are set to 300, 120, and 500 µg/kg, respectively (Johansson and van Bavel 2003a; Canada EPA 2003; Swedish EPA 2002). Also, a standard carried out by Dutch shows the target value as 1,000 µg/kg for 10 PAHs (naphthalene, anthracene, phenanthrene, fluoranthene, benzo(a)anthracene, chrysene, benzo(a)pyrene, benzo(g,h,i)perylene, benzo(k)fluoranthene, and indeno(1,2,3-c,d)pyrene) of unaffected soil and 4,000 µg/kg for soil that required restore (Van Brummelen et al. 1996). Thus, the content of PAHs for fly ash in this study has not exceeded the limits of sensitive land use, but exceeded the limit of Dutch EPA for about 2 times. Therefore, integrated utilization of the fly ash in this study should be performed with caution.

Index toxic equivalent quantities (TEQ) associated with PAHs are used to evaluate the potential health risk of PAHs to ecosystems and human beings. Value of TEQ_i was calculated for each PAH according to the content in the sample and its toxic equivalency factor from the literature (Nisbet and LaGoy 1992). The total TEQ value was calculated by $TEQ = \sum(TEQ_i) = \sum(C\ PAH_i \times TEF\ PAH_i)$, and the results are listed in Table 4. From the study of Nisbet and Lagoy, benzo(a)pyrene and Dibenz(a,h)anthracene are the two most potent carcinogenic PAHs, although they were not detected in the current study. The total TEQ value was 4.00 µg/kg, which is lower than the value of 15 µg/kg for soil by USEPA (2012).

4 Conclusion

The average particle size of discharged fly ash from the incinerator is 9.85 µm, ranging from 0.079 to 30.67 µm, which accounting for more than 90 wt% of the total ash mass. The collected fly ash has only a minimal specific surface area of 3.92 m^2/g and a negligible porosity. Chemical characteristics, using XRF and ICP-MS, revealed the most abundant component is SiO_2 and Pb is a trace element in the highest content with the value of 35,037.90 µg/kg. Fly ash in this study presented a different levels and patterns of PAHs, for having lower content of total 16 PAHs, and mainly consists of small PAHs, comparing with HW ash. These differences would contribute to the combustion temperature and the high content of lead in solid waste. The amount of PAHs in fly ash did not exceed the guidelines for sensitive land use. However, the ash should be regulated carefully before disposition for the high content of lead.

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